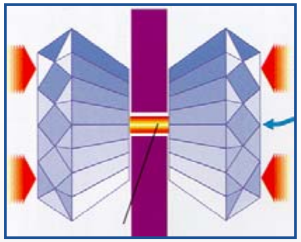
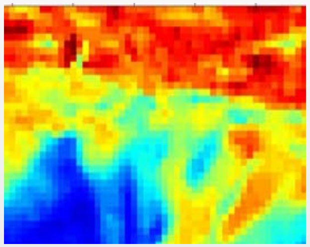
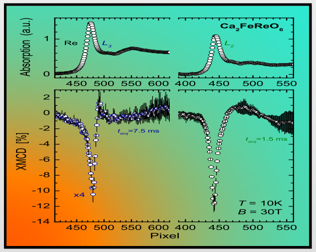
EDXAS workshop
2-5 February 2009
ESRF – Grenoble - France

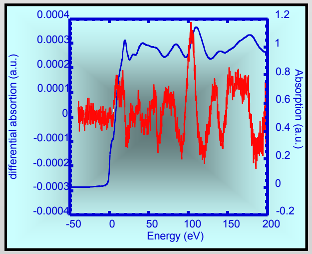
TE-XAS project
Scientific opportunities and technical challenges

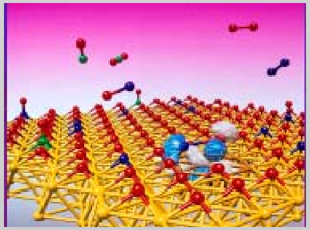
Olivier Mathon, Trevor Mairs, Sakura Pascarelli











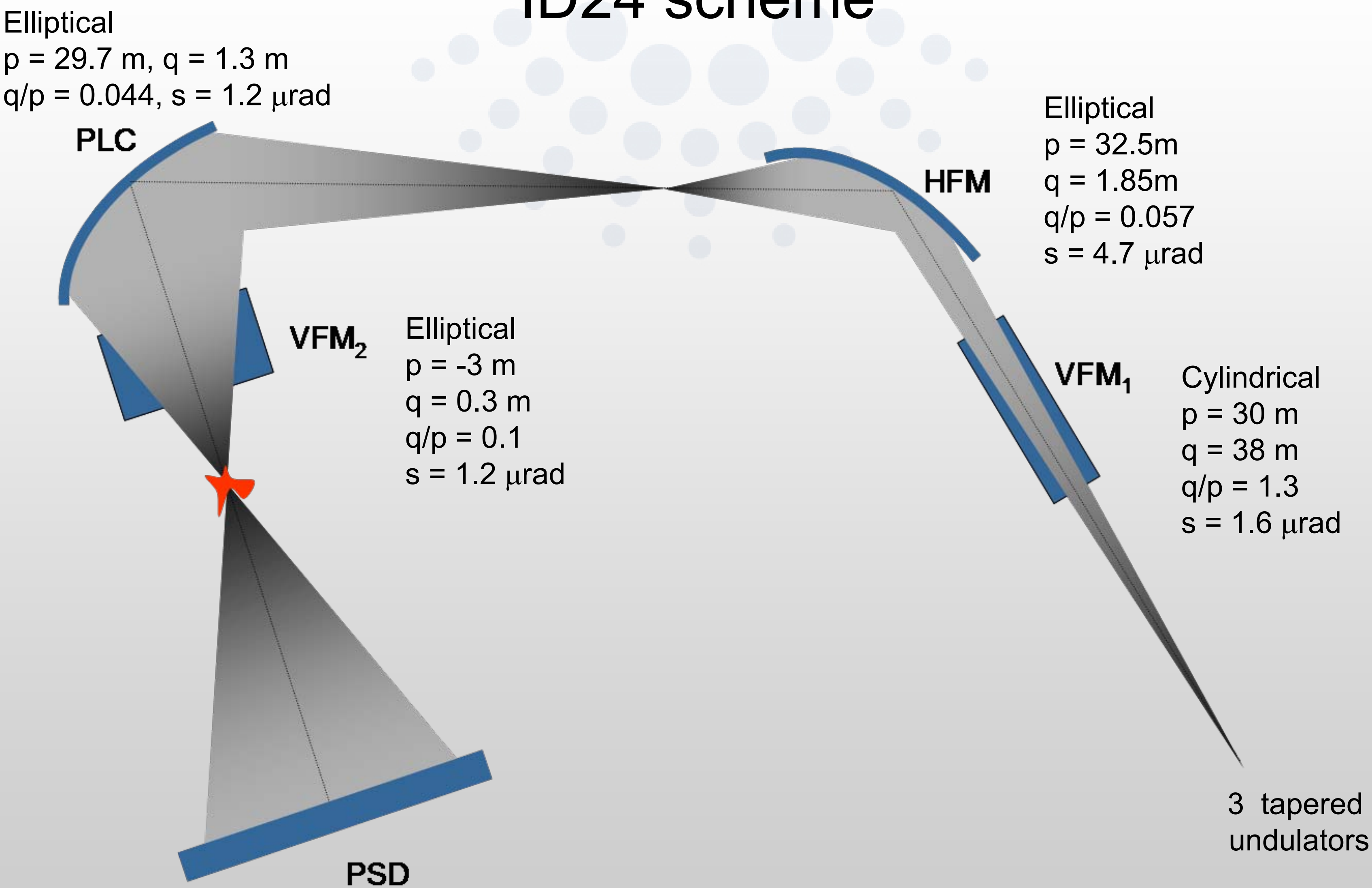
Overview

I. ID24 performance and limitations
- ID24 scheme
- Limitations

II. TE-XAS Project
- General presentation
- Rationale of TE-XAS
- Conceptual design

III. Technical Challenges
- Sources
- Optics
- Design and Stability
- Detection

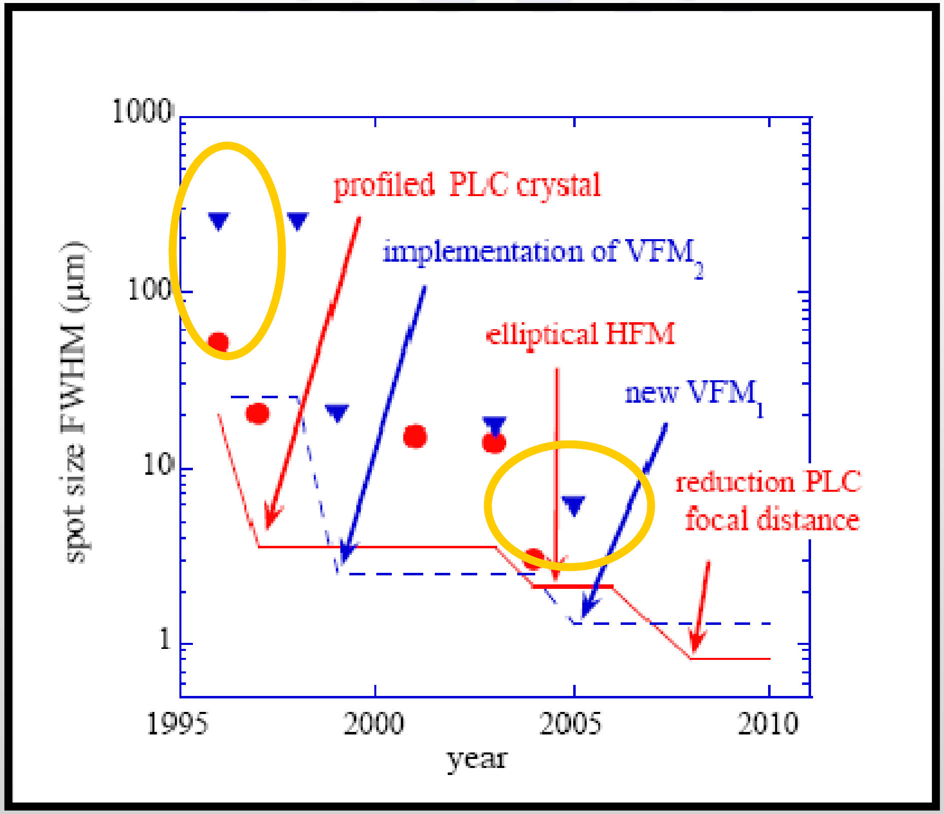
ID24 scheme



Pascarelli *et al*., JSR **13**, 351 (2006)

Evolution of spot size in last 10 years on ID24

ID24 started operation with spot sizes of 50 x 250 μm^2



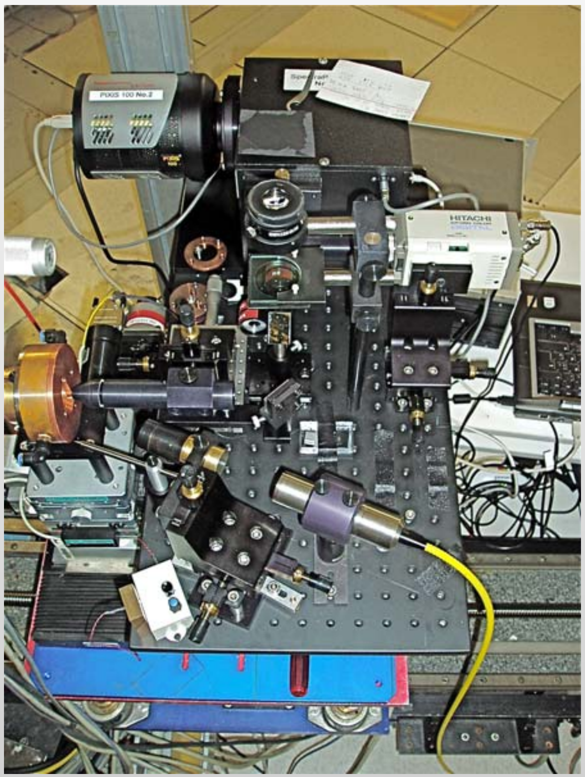
Now spot sizes (low E) are ~ 5 x 5 μm^2

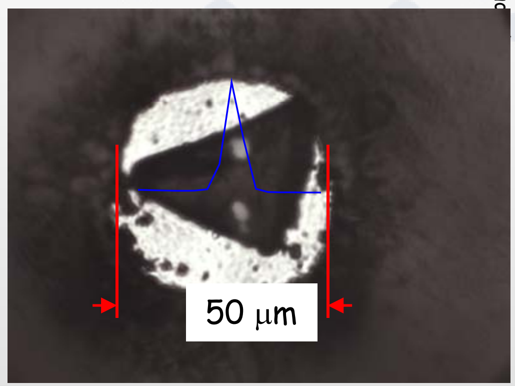
Is a smaller spot size interesting ?

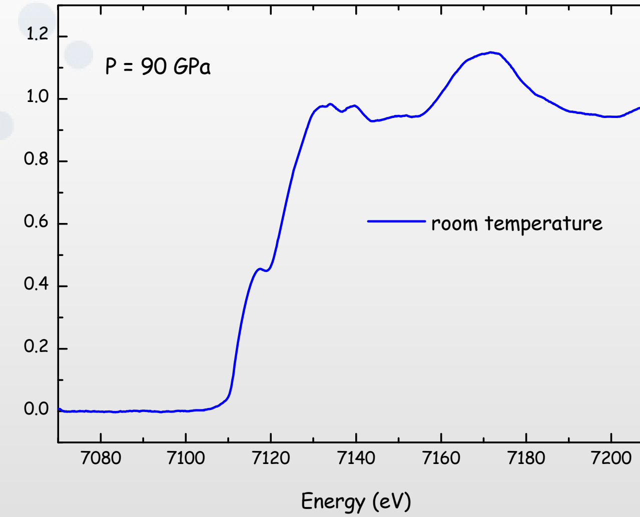
Melts at Megabar pressures

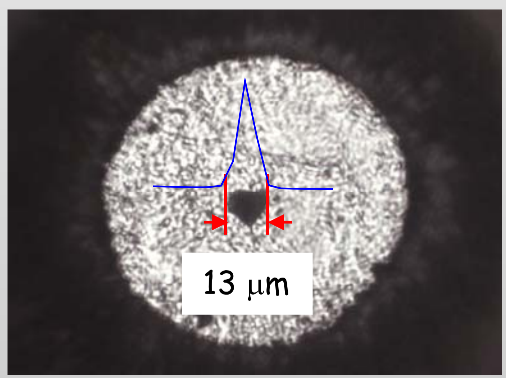
Structure of melts as well as thermodynamic conditions of melting under high pressure are of great interest in different fields: fundamental physics

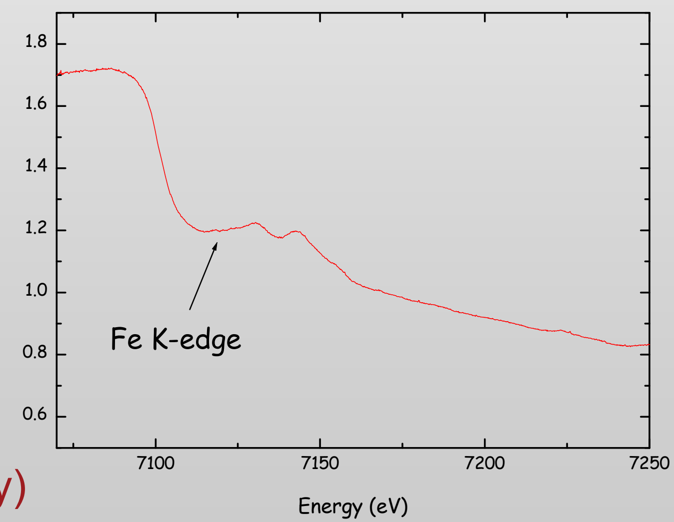
planetary and Earth Science











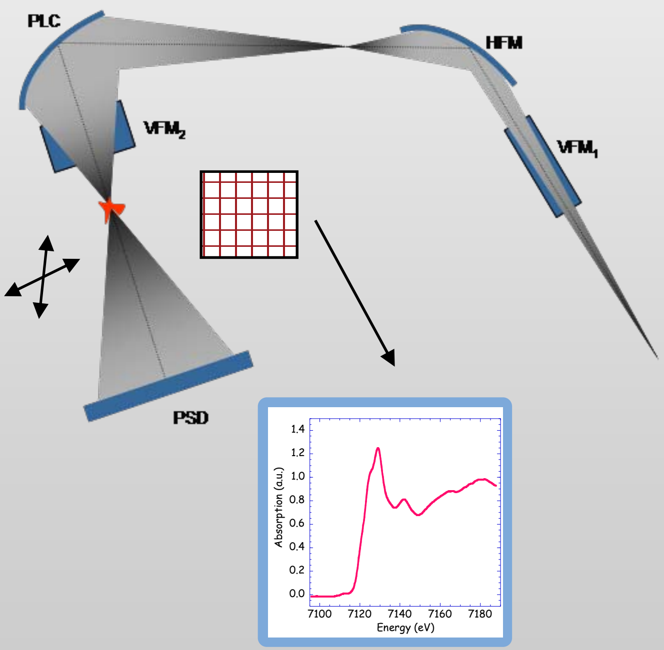
G. Aquilanti
Collaboration with Reinhard Boehler – MPI, Mainz (Germany)

Hyperspectral mapping

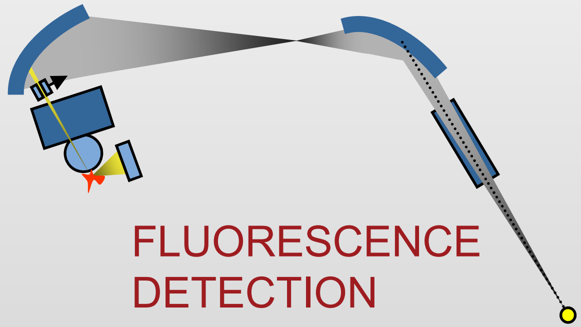
Energy dispersive absorption spectroscopy for hard-X-ray micro-XAS applications
S. Pascarelli *et al.*, J. Synch Rad. **13**, 351 (2006)

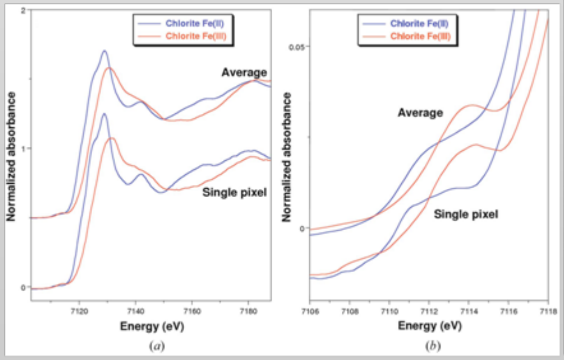
Micro-XAS two dimensional mapping

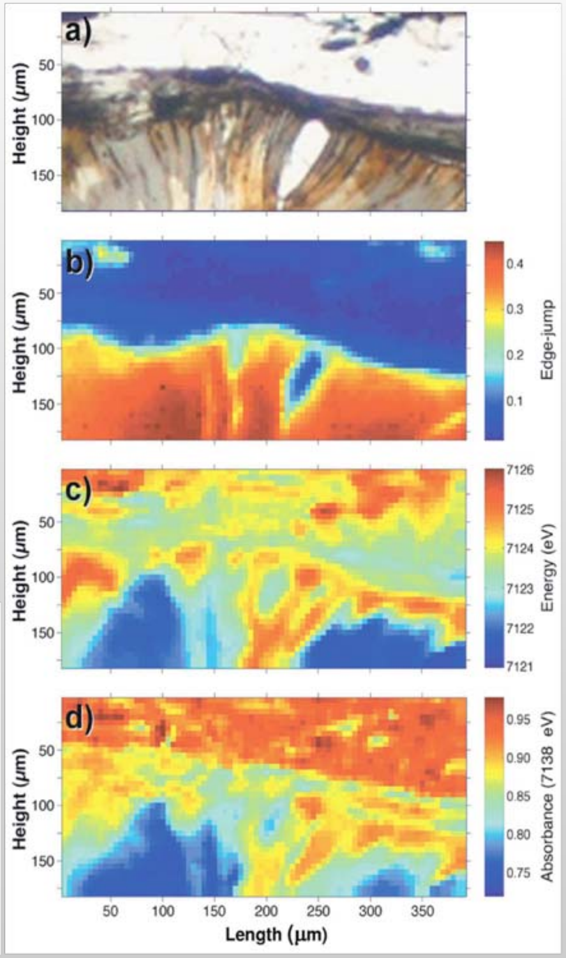
- novel application of EDXAS
- it exploits the main strengths of EDXAS:
  - stability
  - small beam
  - speed



Redox and speciation micromapping using dispersive X-ray absorption spectroscopy: Application to iron in chlorite mineral of a metamorphic rock thin section
M. Muñoz *et al.*, GGG **7**, Q11020 (2006)

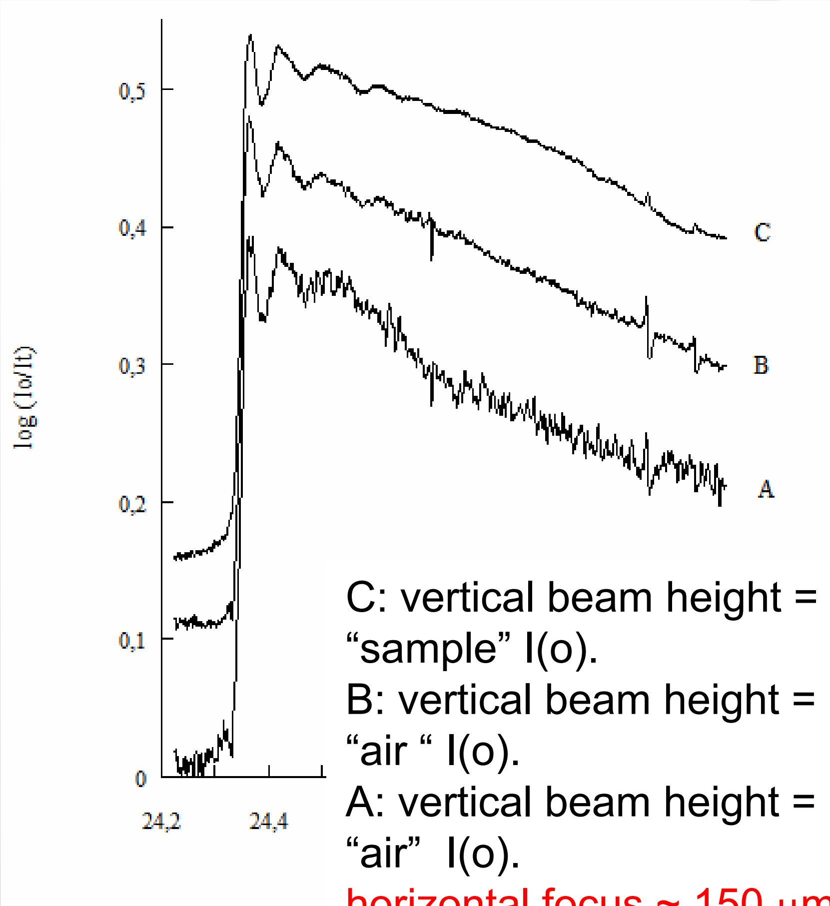






ID24 upgrade : Is a small focal spot always good ?

No !

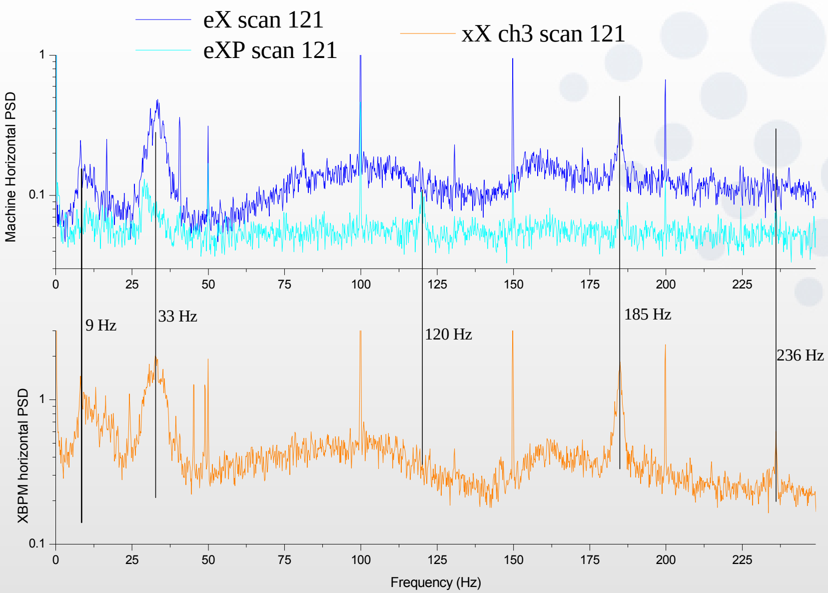


C: vertical beam height = 300 μm "sample" I(o).
B: vertical beam height = 300 μm, "air " I(o).
A: vertical beam height = 100 μm, "air" I(o).
horizontal focus ~ 150 μm

Ever smaller beams make this sort of experiment ever more difficult to realise: *even now we cannot achieve this performance at <15 keV with Bragg polychromators because of this*.

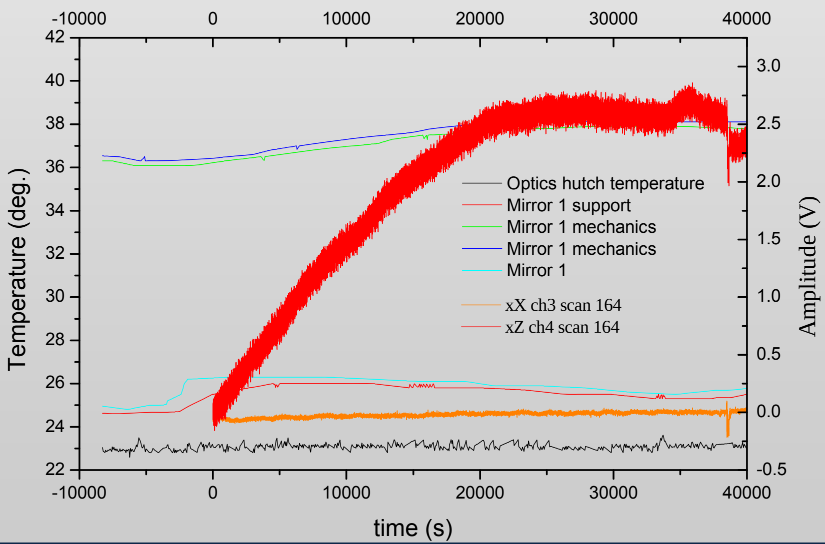
M. A. Newton, J. Synchrotron Rad., 2007, **14**, 372-381.

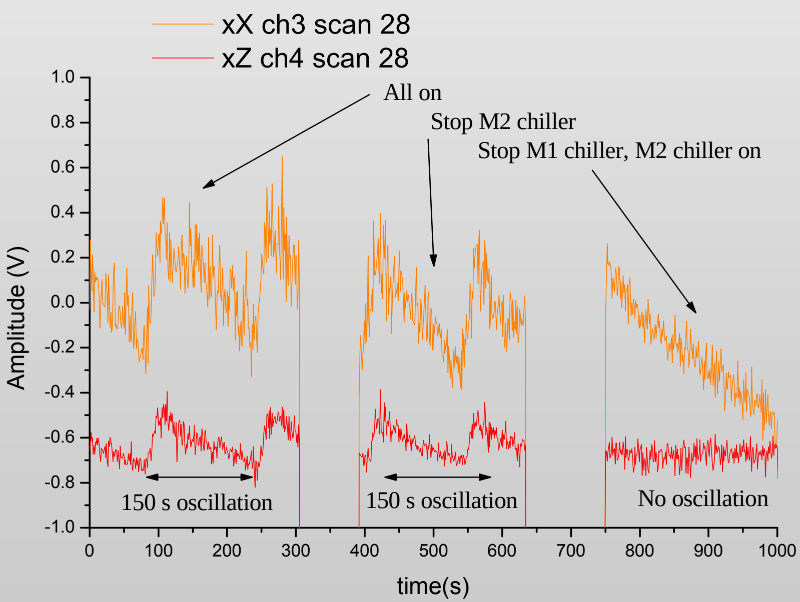
Stability



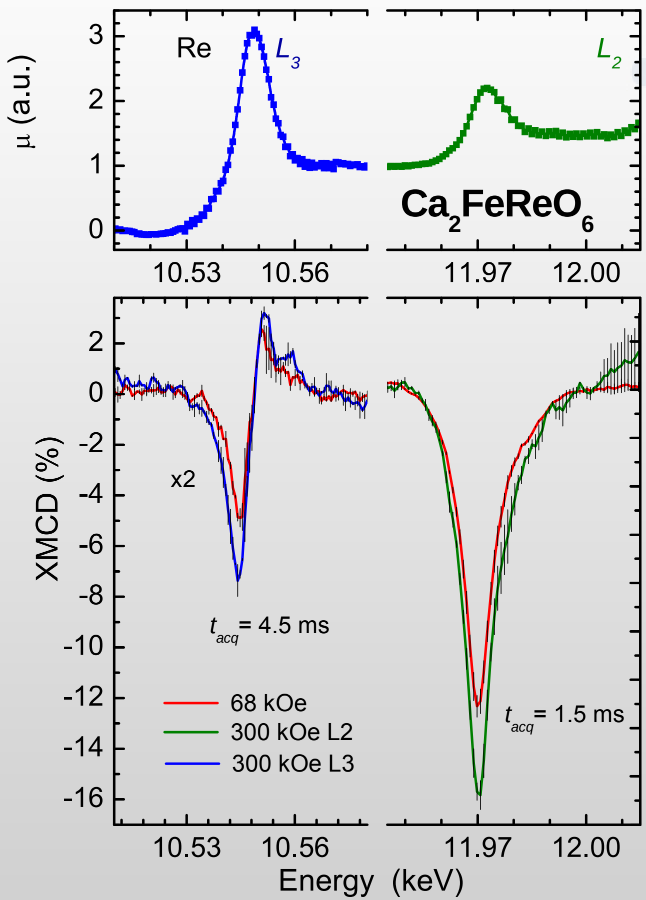
Main effects

- heat load control
- temperature drifts of the optical element
- temperature stability of the hutch
- mechanical vibration
- stability of the electron beam

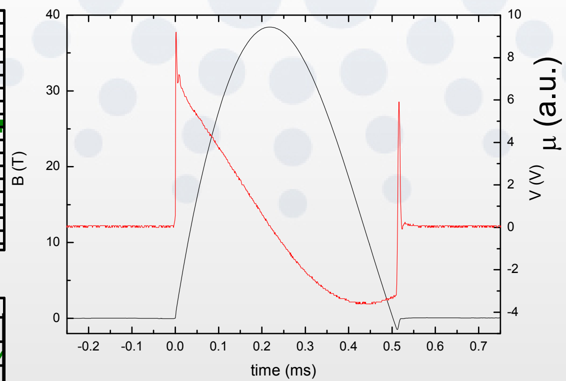


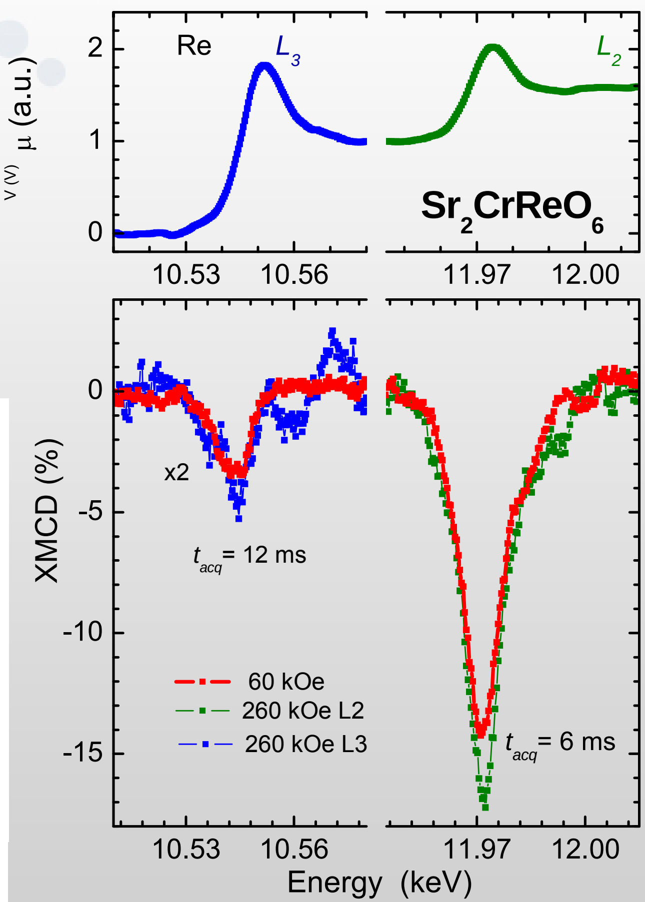


XMCD under high magnetic fields



XH detector





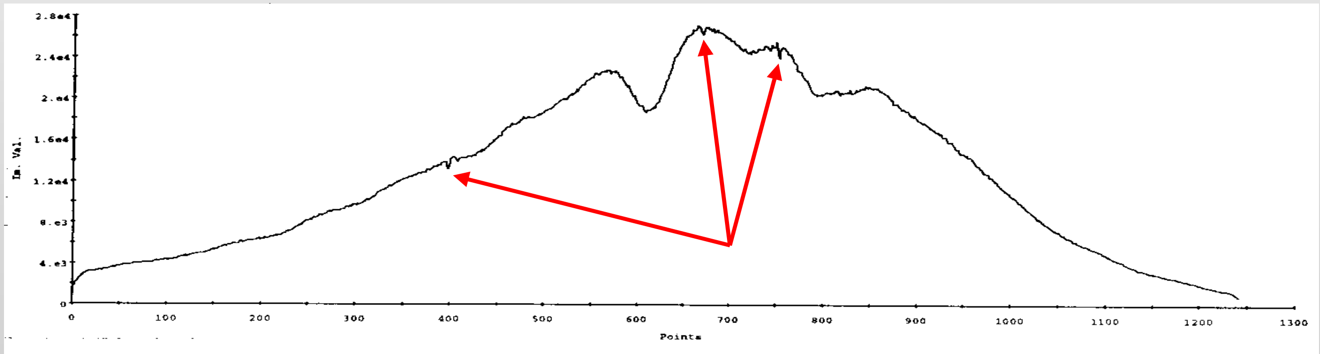
gated FReLoN detector

M. Sikora, in preparation (2009)

Crystal degradation

Polychromator crystal in Bragg case under X-ray beam degrades

- not visible on new crystals
- appears quickly under X-ray beam exposition
- appears more quickly at low energies (7 keV)
- are localized
- depends on the Bragg reflection: (111) more sensitive than (311) or (220)
- not visible with topography, microscopy



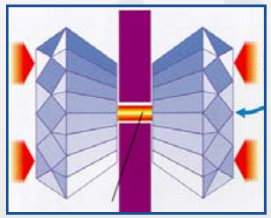
Polychromator under high vacuum (F. Baudelet experience at SOLEIL)

We need to upgrade because:

1. The decrease of the spot size has opened new scientific opportunities:

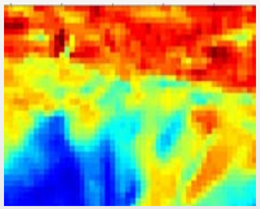
Explore Earth's core

- **"in-situ" laser heated DAC**
- **spatial or time resolution**



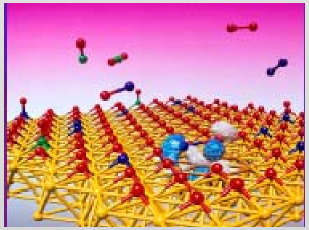
Hyperspectral mapping

- **full EXAFS/pixel**
- **rapid**



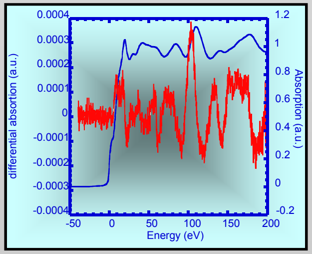
BUT, the full exploitation of a 2x2 μm^2 spot requires a new BL design based on new stability and precision standards

2. The drastic decrease of the spot has penalized all activities on samples heterogeneous on the micron scale (i.e. heterogeneous catalysts or magnetic materials for "real" devices)



We are <u>unable</u> today to measure EXAFS on some of the most interesting systems (i.e. atomic scale magnetostriction in Terfenol-D).

→ large spot required to average over spatial heterogeneities



Time resolved and Extreme conditions XAS

Time resolved

ms
heterogeneous catalysis
cleaner chemical processes
emission free vehicles
new energy resources

µs
solution chemistry
photochemistry
microfluidics

ns → ps
recombination
radiative decay
rotational motion
strain propagation

fm atomic displacements
reversible H_2 storage processes
energy-driven magnetic materials
magnetostriction, piezoelectricity

kinetics
phase transitions
chemical reactions at HP and HT

fast 2D hyperspectral mapping
redox, speciation

pulsed high magnetic fields

laser shocked matter

Extreme conditions

P > 100 GPa
T > 3000 K

earth and planetary science
speciation
oxidation states
partition coefficients
complexation in aqueous fluids

warm dense matter
equillibrium $\tau \sim \mu s$
plasma opacity
local order

material science
HP properties of catalysts
new materials

melts
electronic structure
local order

What drives our technical choices for the TE-XAS project

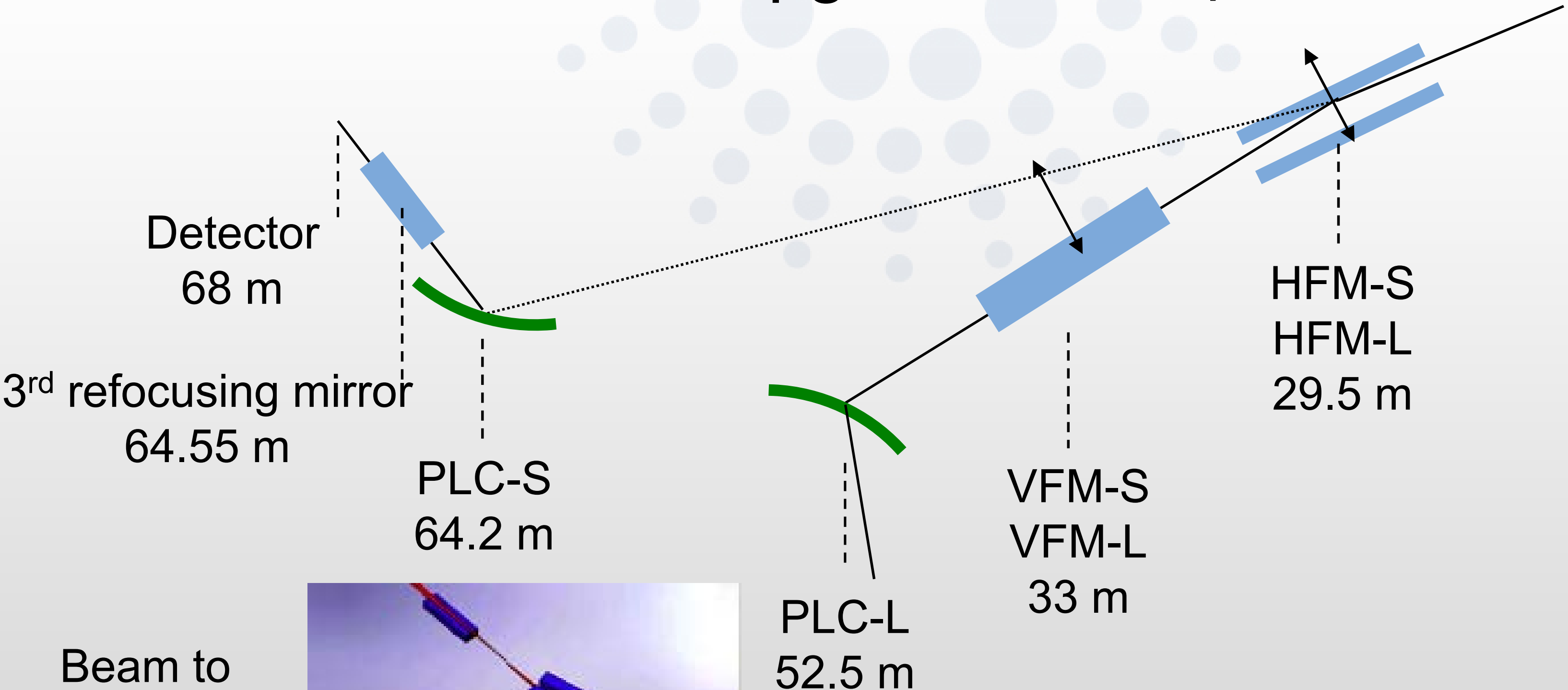
1. No compromise on flux, focal spot, sample environments
2. Synergy with standard EXAFS activity
3. Shared infrastructures ID24/BM29

- Stability
  - Beam stability (Source, vibration)
  - Thermal stability (Laue, mirror, hutches)
  - Setup stability (optimized, stability, manpower and beamtime efficiency → 30% waste)

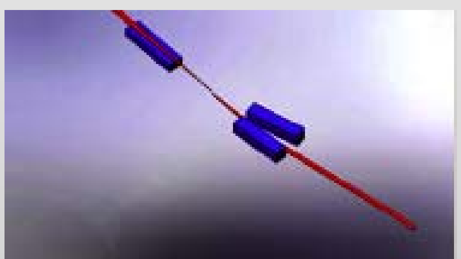
- Optimized Optics
  - EDXAS-S
  - EDXAS-L
  - ΔE extended at low E
  - High flux
  - crystal degradation (UHV)

- Detection
  - ms down to the μs
  - 100 ps in pump and probe mode
  - fluorescence 100 ms
  - ps using streak camera

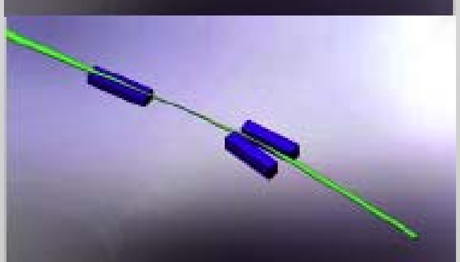
ID24 upgrade : concept



Beam to
TE-XAS S



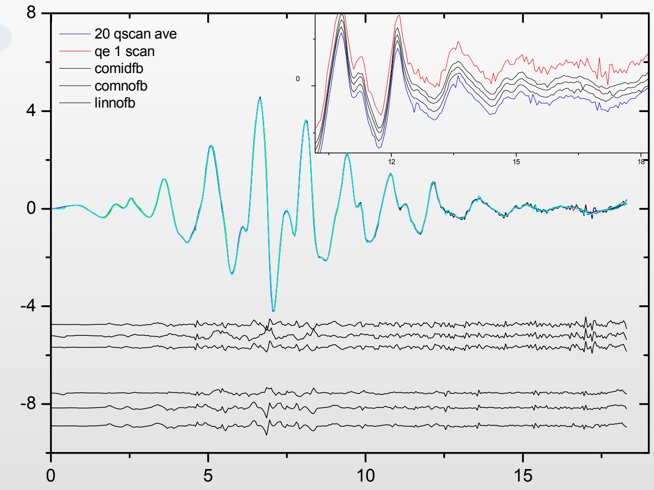
Beam to
TE-XAS L

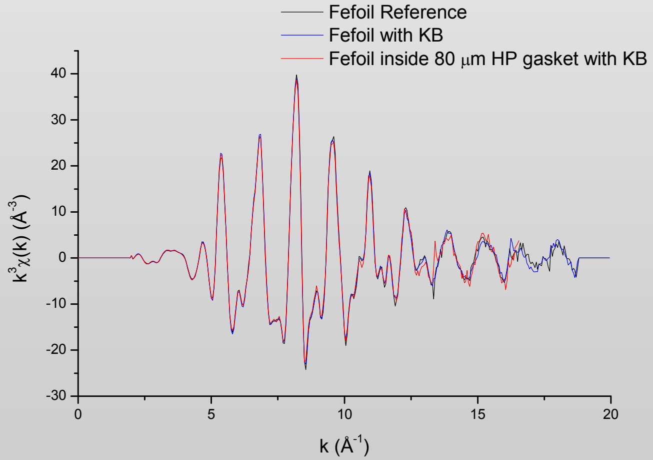


- Optical design / Xray beam optimized
- Hutch and sample environment optimized
- Fixed setup
- Gain in time/efficiency
- Gain in ΔE at low E

EXAFS: a "standard EXAFS" station on BM29

- No fundamental change in the layout
  - Monochromator, mirrors, experimental hutch
  - Transmission with very good signal to noise ratio
  - Fluorescence and TEY
  - Improvement of the stability
- Upgrade of the monochromator
  - New mono with liquid N2 cooling
  - 2/3 pairs of crystal in mono 111/311/511
- Possibility to perform Q-EXAFS scans
  - one scan every second
  - up to k=20 $Å^{-1}$ with good S/N
  - test on a Co foil in 1 s (1)
- μXAS capability ?
  - 3 x 3 μm^2
  - up to k=20 $Å^{-1}$ with good S/N
  - test on Fe foil (2)





(1) Prestipino *et al*., to be published (2008), (2) Ziegler *et al*., accepted to Xray spectrometry (2008)

Source

- BM versus wiggler versus undulator
  - Wiggler : no coupling optics needed
  - Useful Flux (ph/s/0.1%BW), 300 mA

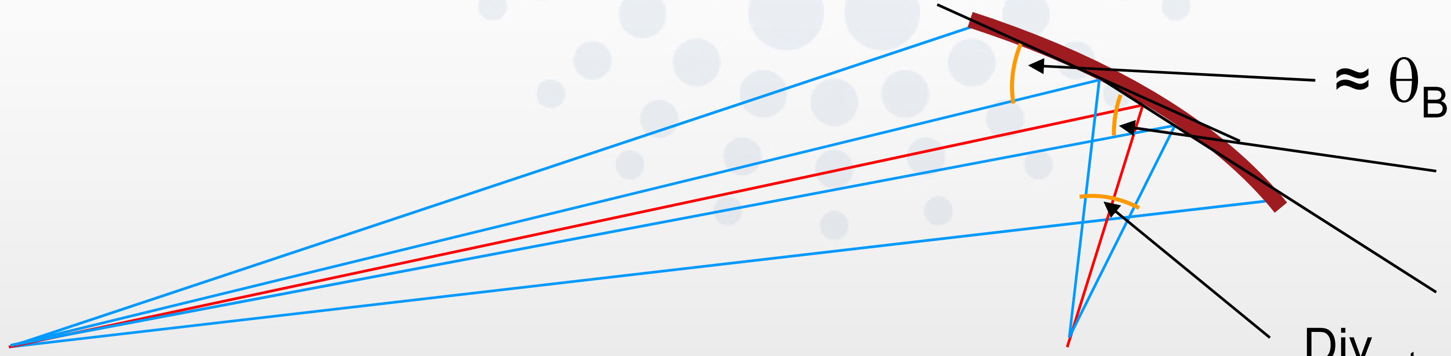
X 20 (BM → W70)
X 5 (W70 → U27/U32)

	7 keV	11 keV	20 keV
BM	4.10^{13}	$3.5.10^{13}$	3.10^{13}
W70	$7.2.10^{14}$	$7.2.10^{14}$	4.10^{14}
U27/U32	$4.5.10^{15}$	$2.5.10^{15}$	$1.1.10^{15}$

  - Total power   Power Wiggler = X 5 Power Undulator

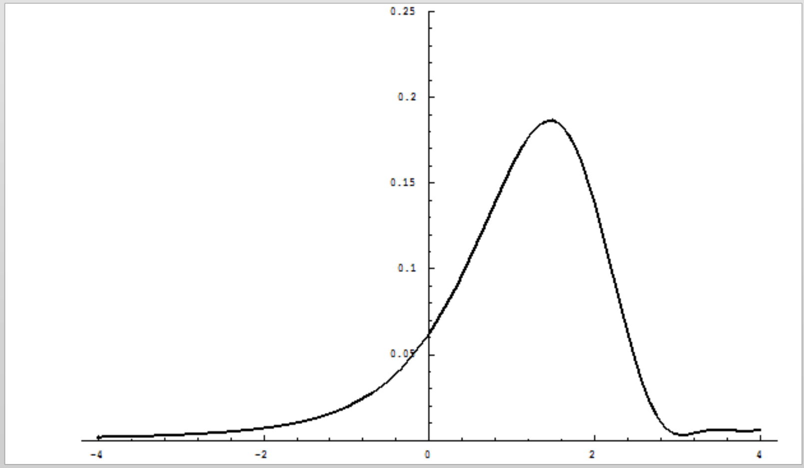
- High β versus low β
  - Low β : smaller source and higher divergence
  - Present low β not sufficiently low to avoid coupling optics
  - No possibility to tune “our” β function
  - If coupling optics is needed, then high β is the best compromise

EDXAS – S : How small can the focal spot be ?



Around the Bragg condition, only a small part of a monochromatic beam is diffracted the divergence of the accepted beam

$\approx \omega_D \approx 40\ \mu rad$ (111, 7 keV)



If diffraction curve is approximated by a rectangle focusing diffraction limit is:

$$\Delta x_{dyn}.Div_{out} = 0.89\ \lambda$$

$$\Delta x_{dyn} = 0.89\ 1.8.10^{-10}/79.10^{-6} = 2\ \mu m\ !!$$

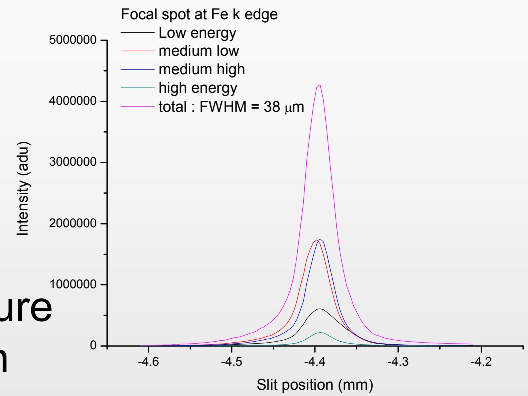
Confirmed by first dynamical simulation of the Bragg case (V. Mocella, C. Ferrero)

EDXAS – L : How large can the focal spot be ?

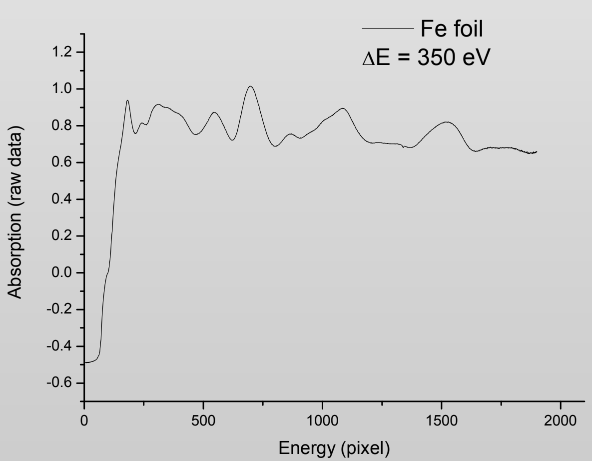
Principle : based on dynamical properties of Laue polychromator case
→ Horizontal focal spot about 40 μm

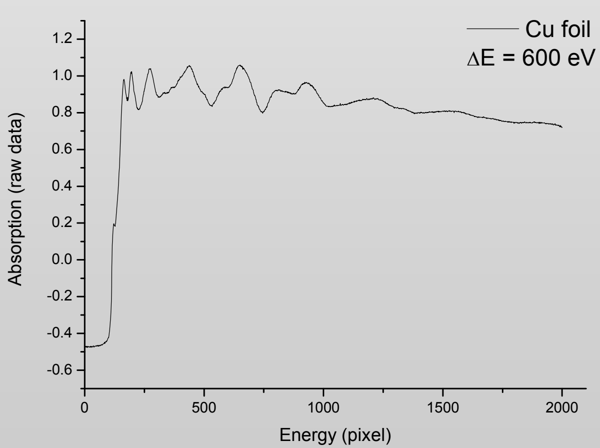
Main difficulty : Heat load on the crystal.

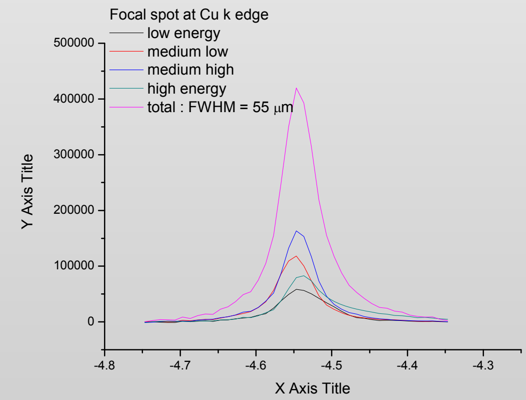
Possible solution : liquid N_2 cooling → dynamical bender impossible
→ Crystal fixed on a Cu block with constant radius of curvature
→ Change q = f(E) and correction of the spherical aberration



First test are encouraging (measurements)
q (7 keV) – q (9 keV) = 20 mm; q (20 keV) – q (25 KeV) = 2 mm
Spherical aberration correction in both cases < 20 μm

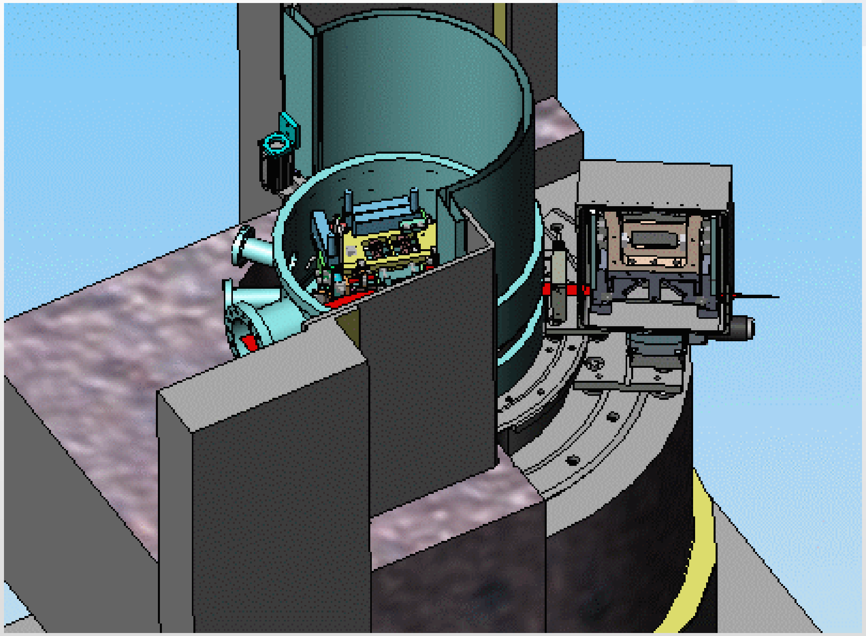


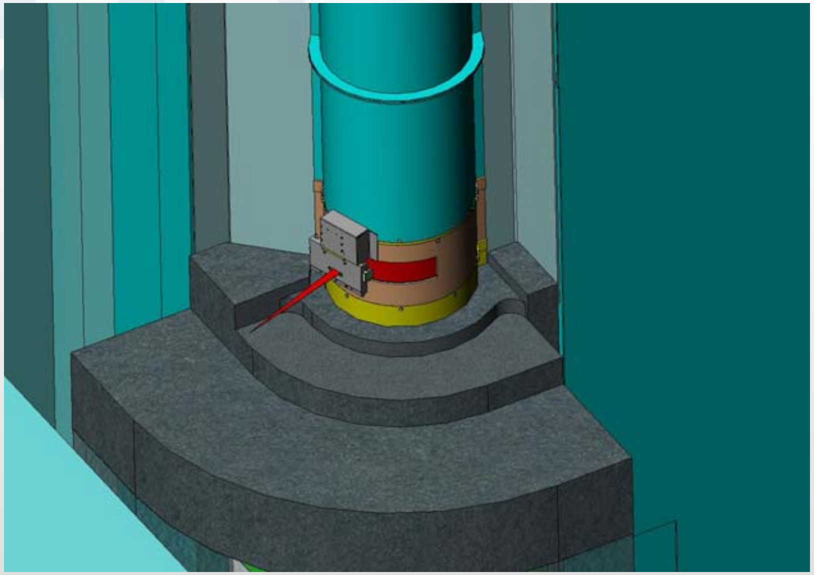


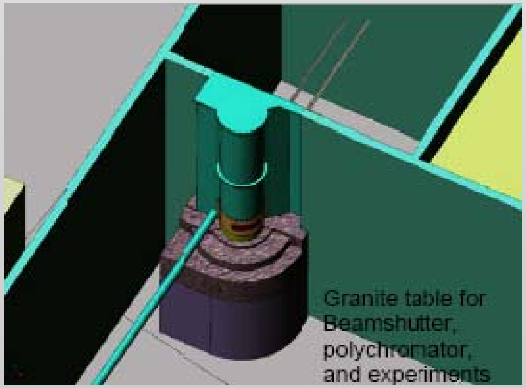


Bhattacharyya D. *et al*, in preparation (2009)

Mechanical Design

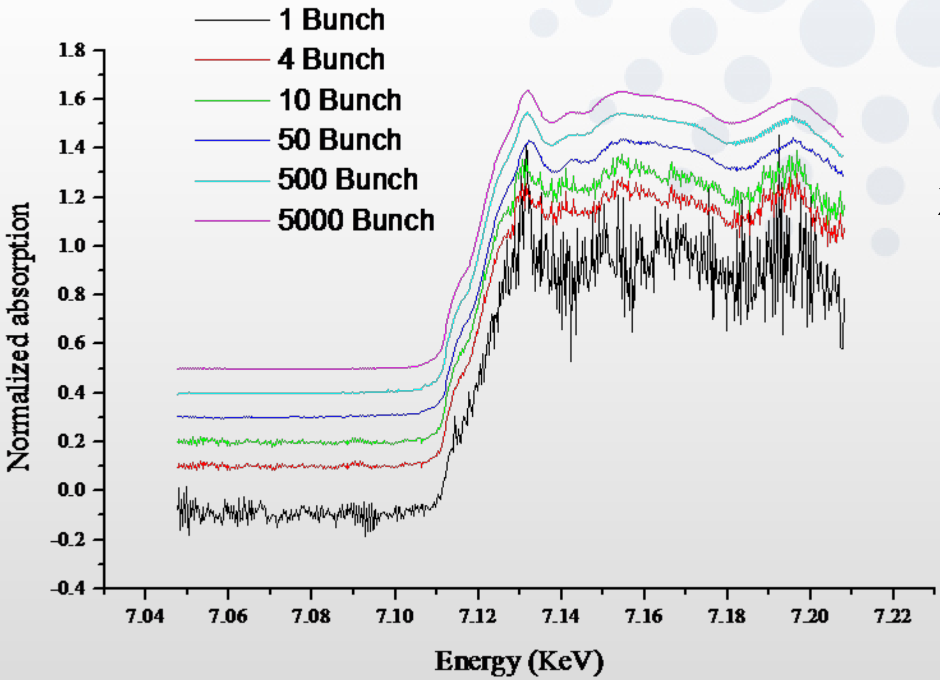






- Cooling white beam mirror
- Mechanics of the facing mirrors
- Bragg polychromator environment
- New Laue bender
- Sample environment
- Safety

Detection



XH at ESRF ID24(1)

- good signal to noise ratio obtained already with 50 single X-ray bunches (4 bunch mode)
- equivalent to 2 μs in multibunch at 300 mA with 3 undulators

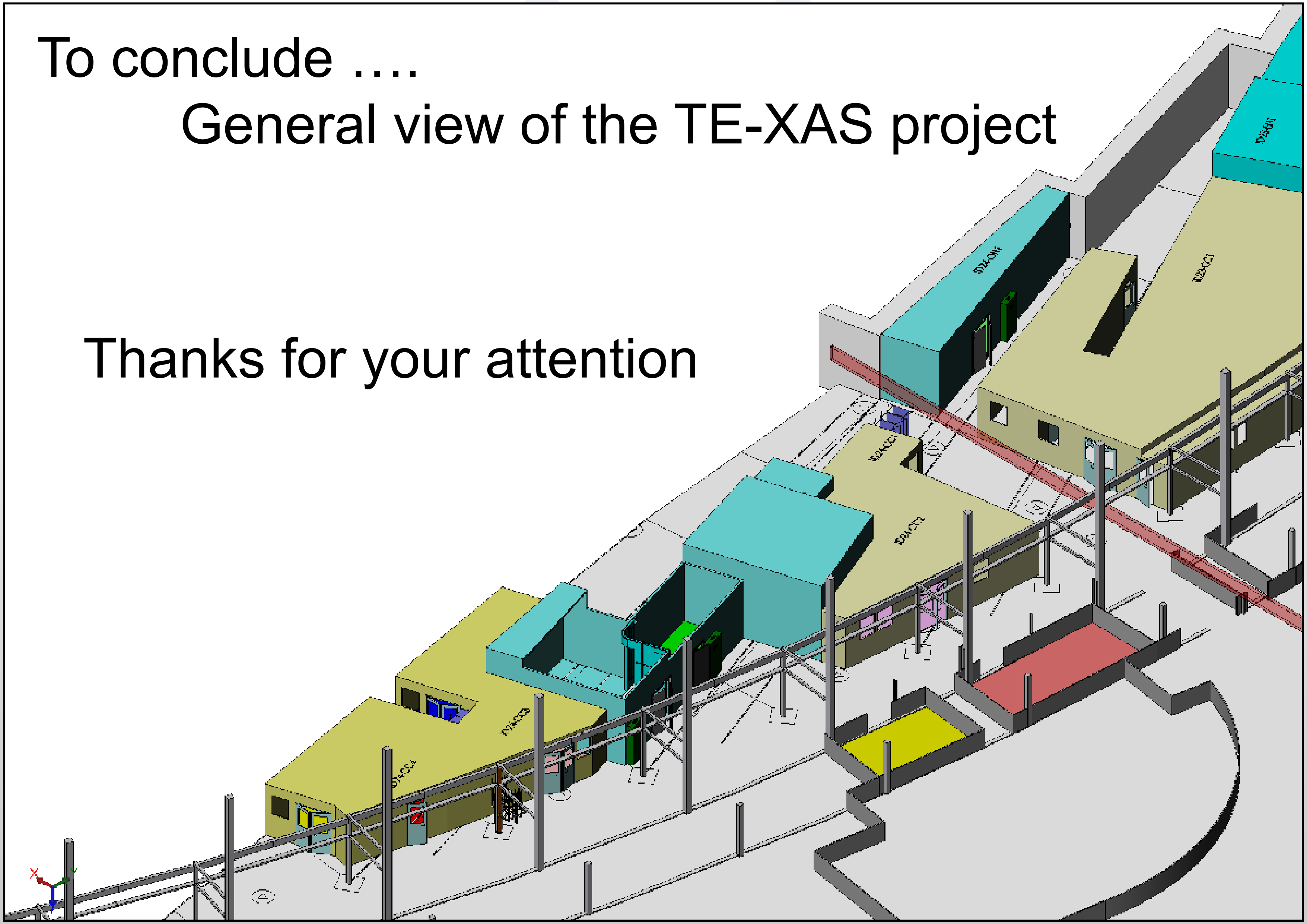
Other detection schemes

- Optimized fluorescence with Turbo scan in 100 ms
- Dispersive setup fully compatible with streak camera concept

(1) J. Headspith et al., *IEEE Nucl. Sci. Cof. Rec.* **N55-2**, 2421 (2007).

To conclude ….

General view of the TE-XAS project



Thanks for your attention